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IS 12378 (1988): Method for Macroetch Test for Titanium Alloys [MTD 22: Metallography and Heat Treatment]

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Indian Standard

METHOD FOR MACROETCH TEST FOR TITANIUM ALLOYS

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Gr 3 *November* 1988

Indian Standard

METHOD FOR MACROETCH TEST FOR TITANIUM ALLOYS

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 18 April 1988, after the draft finalized by the Heat Treatment and Metallography Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Macroetch test method is frequently used as evaluating technique for revealing solidification structure, segregation, general distribution and size of inclusions, porosity, grain size, flow lines, surface condition, manufacturing defects and method of manufacture. It is done by etching a suitably prepared specimen and examining it visually or at low magnification (not exceeding ten diameters). Macroetch test method provides inspection of a large area possible as opposed to microetch test method.

0.3 In the preparation of this standard, assistance has been derived from the following standards:

ASM ED 8 Metal Handbook, Vol. 7

ASM 1978 — Metallographic Etching, G. Pettov

ASM 2642 - Etch Anodise Inspection Procedure

AD Mcquillon and M.K. Mcquillon Butts worth Scientific Publication, London 1956

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1. SCOPE

1.1 Macroetching is the etching of specimen for examination at low magnifications and is used to reveal the macrostructure of titanium alloys products, such as bar, billets, forgings and castings. The present procedure is intended to be followed for classifying titanium alloys by a graded series showing the characteristic structure and incidence of certain processing defects. This method is limited in application to titanium alloy billets, forgings and castings.

2. SIGNIFICANCE

2.1 Macroetching is a simple test which provides information about the heterogeneity of the sample. It will show: (a) variation in structure such as grain size and columnar structure; (b) presence of acute segregation and banding, and (c) the presence of discontinuities, such as laps, seams and bursts.

3. SAMPLING

3.1 When using macroetching as an inspection procedure, sampling should be done in an early stage of manufacturing so that if the material proves defective, the minimum amount of unnecessary work is done. However, the sample should not be taken so early that further working can introduce serious defects. The sample is usually taken after ingot breakdown and after most chances of bursts have passed. Billets or blooms going into small sizes are sampled after initial breakdown. Materials going into forging billets or other products are sampled at near finish size. Sampling may be done systematically on a random basis.

3.1.1 Some common methods of sampling are listed as follows.

3.1.1.1 *Billets, blooms and hot-rolled products* — Discs are usually cut from these products near the end but fish tails should be avoided. Discs from large blooms can be cut into smaller pieces.

3.1.2 *Forgings and Extrusion* — Discs, cut transverse to the long dimension, are used to see bursts, voids, etc. Forging can be cut parallel to long dimensions to show flow lines. Macroetching of an unprepared specimen shows surface defects, such as shuts, flats, seams, etc.

3.1.3 *Sheets and Plates* — While looking for defects in sheets, as large a sample as possible should be taken. When seeking information on lamination, a transverse section is used.

3.1.4 *Weldments* — A disc perpendicular to direction of welding shows weld penetration, heat affected zone, structure, etc.

3.1.5 *Castings* — Specimen should be cut from different regions depending upon the features sought.

3.2 When macroetching is used to solve a problem, then the problem itself largely dictates the sample location on the work piece and the stage of manufacture; for example, when looking for pipe, the sample should represent the top to the ingot, or when looking for burst, the sample should be taken as soon after hot working as possible.

3.3 Sample may be cold cut by any convenient means. Saw and abrasive cut off wheels and EDS cutting are particularly effective. Since heat generation during abrasive cutting may affect the structure of the sample, the surface to be examined should be at least 10 mm away from cut surface in case of EDS cutting and 3 mm in case of abrasive cutting.

4. SPECIMEN PREPARATION

4.1 Sample preparation need not be elaborate. Any method of presenting a smooth surface with a minimum amount of cold work will be satisfactory. Discs may be faced on a lathe or shaper. The usual procedure is to take a roughing cut, then a finish cut. This will generate a smooth surface and remove cold work from prior operations. Grinding may also be employed using a fine wheel and light finished cuts. When fine detail is required, the specimen should be finished with metallographic paper.

4.2 After surface preparation, the sample shall be cleaned carefully with suitable solvents. Any grease, oil or other residue left on the surface will produce uneven attack during macroetching. Therefore, care should be taken not to touch the sample surface or contaminate it in any other way after it has been cleaned.

5. SOLUTIONS

5.1 The most common solutions for macroetching titanium alloys are given in Table 1.

5.2 Caution must be observed in mixing the acids as they are strong and can cause chemical burns. The acid should be added slowly to water. Mixing and etching should be done in a fume hood.

6. PROCEDURE

6.1 The specimen may be etched by immersion or by swabbing or by both means. The time of etching will vary depending upon the composition, size and structural condition. Generally speaking 30 s to 10 min will be sufficient. Over-etching can lead to misinterpretation and hence light etching is better. Table 1 gives the time for various etchants, but should be used as a guide only. After etching, specimen should be washed in running water and blown dry with compressed air.

7. EXAMINATION OF SPECIMENS

7.1 Any special condition like segregation, banding, voids, etc, that can be observed should be reported. A few examples are given in Fig. 1 to 5. A low magnification (X 10) stereo microscope can be used for observation. Normally it is essential to see the surface from various angles to observe flow defects and banding. The macrograin size for forged billets can be compared with macrostructures given in Fig. 6 and level of grain size reported.

7.2 The acceptance and rejection criterion for these macro-structures has to be worked out according to the type of alloy and application. Therefore, the basis of macroetching inspection will be a matter of agreement between the manufacturer and the purchaser, if this technique is being used as a quality test.

TABLE 1 ETCHANTS FOR MACROETCHING OF TITANIUM ALLOYS

Sl No.	Etchant	Etching Procedure	Remarks
1.	Kroll's etch (HF 10 ml HNO_3 30 ml H_2O 50 ml)	Swabbing or immersion at room temperature for 30-60 sec	Ti and Ti alloys (high alloying elements)
2.	HF (40 percent) 10 ml H_2O_2 (30 percent) 60 ml H_2O 30 ml	Swabbing at room temperature until desired contrast is obtained. Rinse in cold water and dry	Used for iodide titanium
3.	HF (40 percent) 10 ml HNO_3 40 ml H_2O 50 ml	Immersion at 60-80°C for 5-8 min	Used for Ti and Ti alloys (low alloying elements)
4.	HF (40 percent) 2 ml H_2O 200 ml Iron nitrate 10 g Oxalic acid 35 g	Immersion at 50-60°C from 10 sec to 1 min	For welded seams in titanium alloys and for alpha titanium alloys
5.	HCl 50 ml H_2O 50 ml	Immersion at room temperature from 30 sec to 10 min	Differentiate alpha and beta titanium
6.	HF 4 ml HNO_3 20 ml H_2O 70 ml	Immersion at room temperature from 30 sec to 2 min	Reveals beta forged structure
7.	HCl 20 ml HF 40 ml H_2O 40 ml	Swabbing or immersion in a solution at 50-70°C. Rinse until desired contrast is obtained. If smut forms, immerse in 30% H_2SO_4 for 3 min. Rinse and dry	Desmutt Ti-13V-11 Cr3Al alloys. Others do not normally need desmutting
8.	HF 8 ml HNO_3 10 ml H_2O 82 ml followed by NH_4HF_2 in H_2O (18 g/l) (using fresh solution)	Immersion at 45-50°C for 2-3 min and rinse in cold water Immersion in agitated bath of solution at room temperature for 1-2 min and clean in hot water (90-100°C)	
9.	H_2SO_4 7-8 ml HF 4 13 ml Urotripine 5-6 ml water 100 ml	Swabbing or immersion in solution at room temperature until desired contrast is obtained	

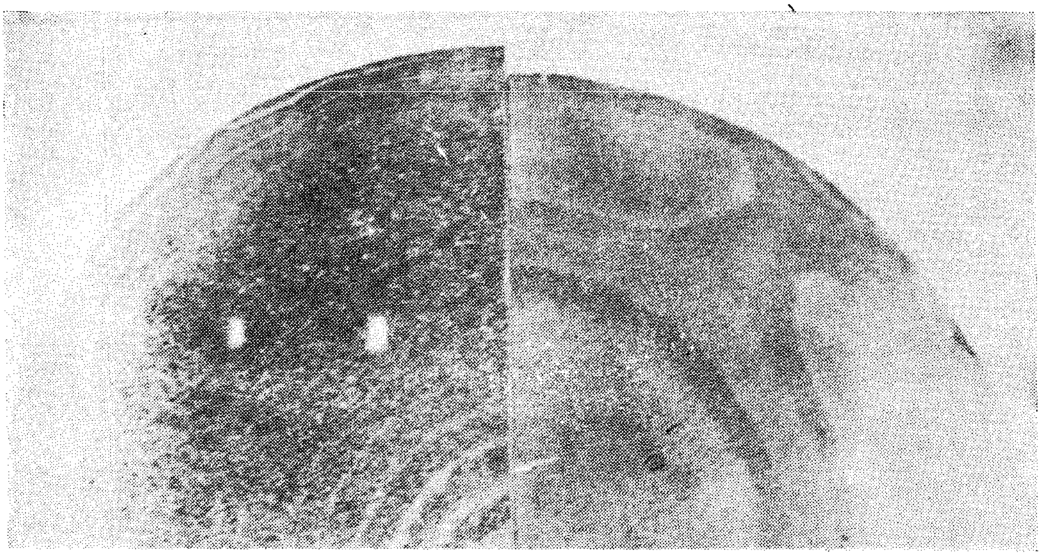

Fig. 1 Ti←6Al←2Si←4Zr←2Mo, $\alpha+\beta$ Processed Billet Illustrating "Tree Rings" Which Represent Minor Compositional Fluctuations During Melting. The Slices are from Two Different Locations. Etchant : Not Known

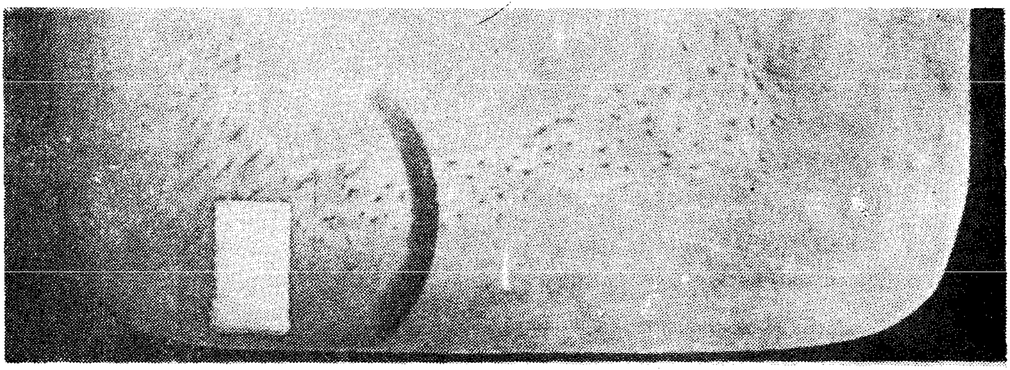

Fig. 2 Ti←6Al←6V←2Sn, $\alpha+\beta$ Processed Appearance of Beta Flecks That Appear as Dark Spots. Etchant (1) 8 ml HF, 10 ml HNO_3 and 82 ml H_2O then 18 ml of NH_4HF_2 in Water

Ref. Metals Handbook, Vol. 9, 1985.

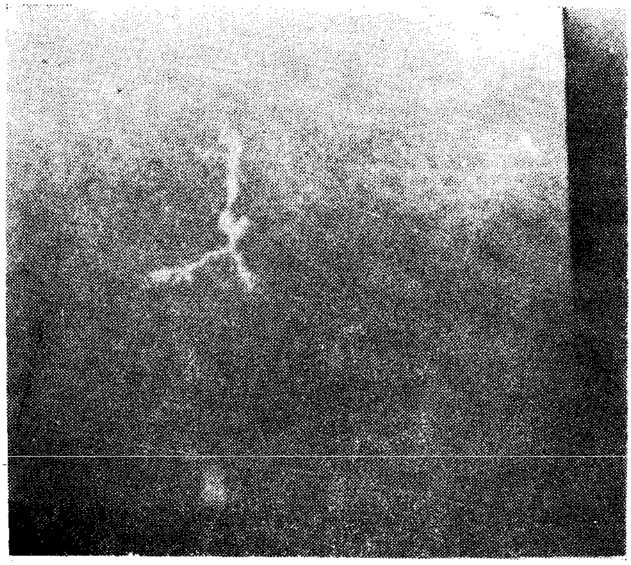

FIG. 3 Ti←6Al←4V, $\alpha+\beta$ PROCESSED BILLET ILLUSTRATING MACROSCOPIC APPEARANCE OF A HIGH INTERSTITIAL DEFECT × 1

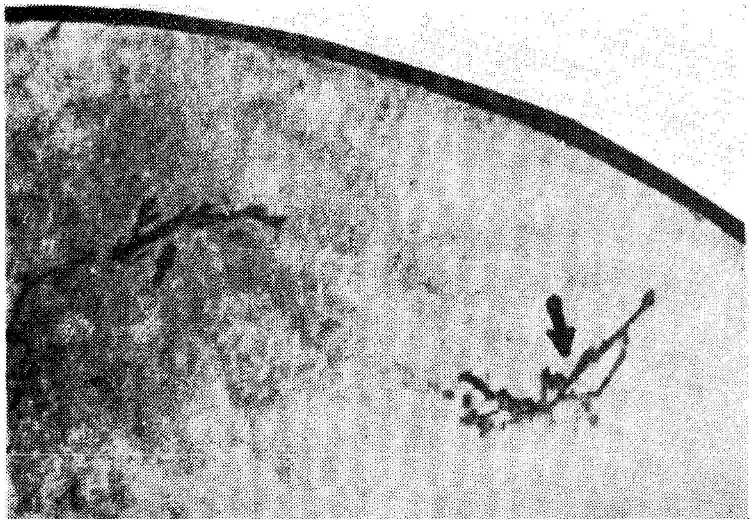

FIG. 4 Ti←6Al←4V, $\alpha+\beta$ PROCESSED BILLET ILLUSTRATING APPEARANCE OF HIGH ALUMINIUM DEFECT × 1·25

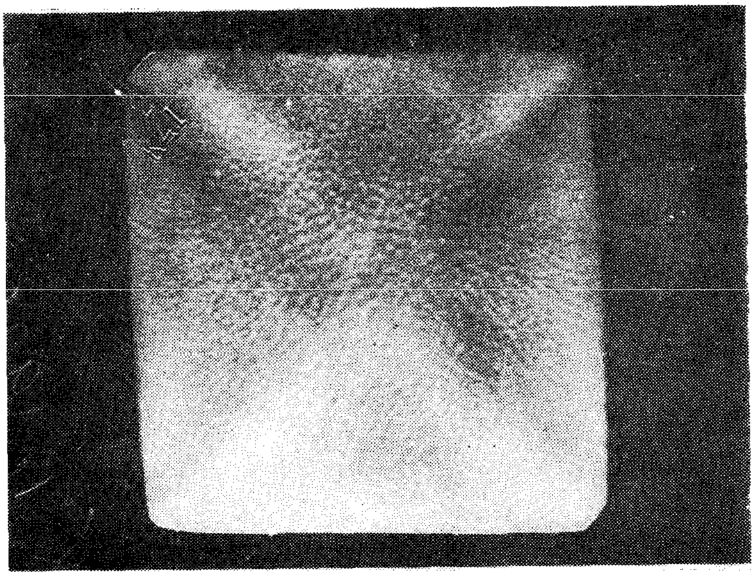

FIG. 5 FORGING SHEAR BAND IN α--β PROCESSED BΓ←9 ALLOY

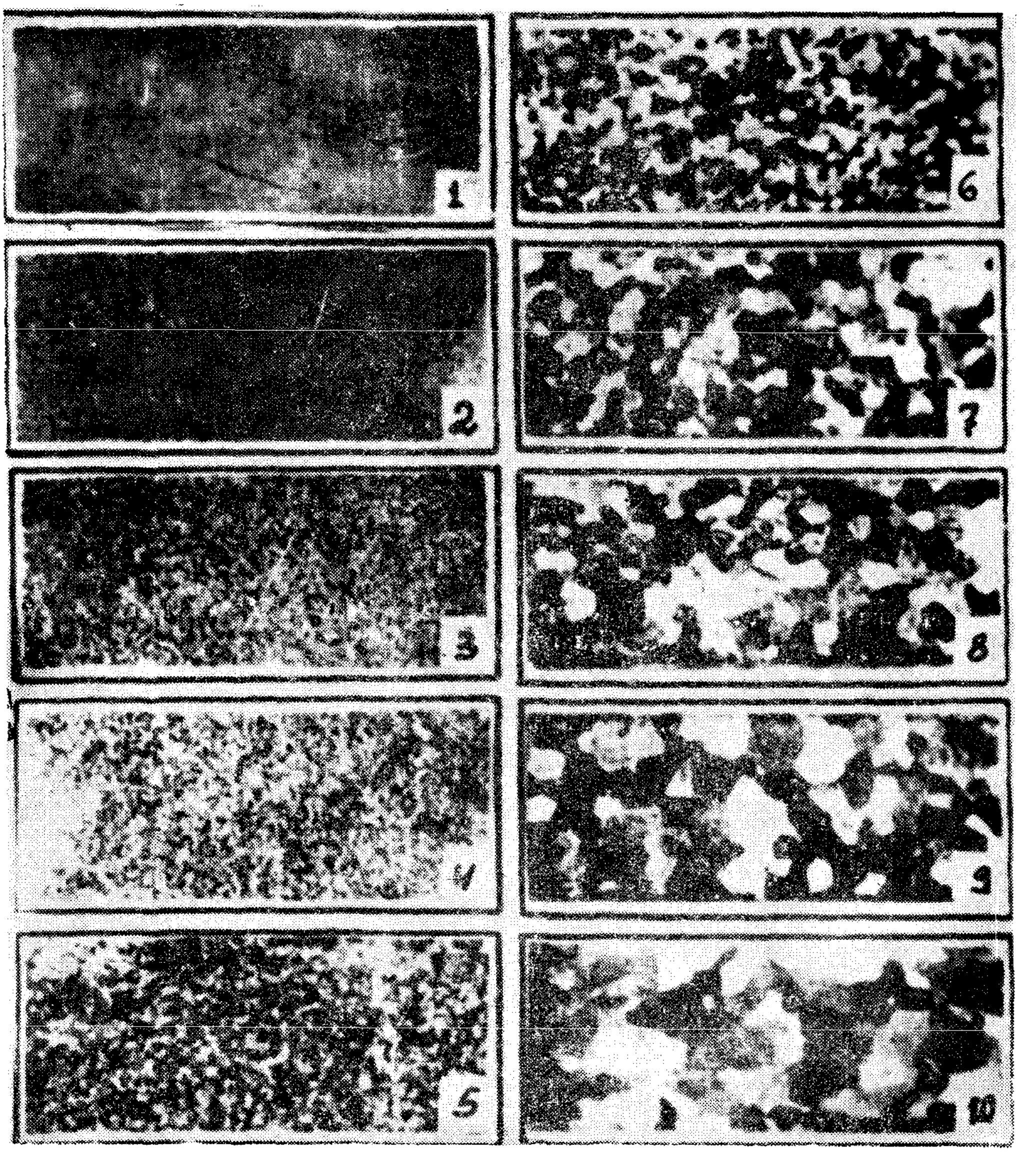

Fig. 6 10 Point Scale of Macrostructures for Comparison of Grain Size Levels for Forged Billets